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On Pins and Needles: Tracing the Evolution of Copper-base Alloying at Tepe Yahya, Iran, via ICP-MS Analysis of Common-place Items

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From 1967 to 1975 a team of archaeologists excavated the site of Tepe Yahya in southeastern Iran under the direction of C. C. Lamberg-Karlovsky. Although there are two forthcoming "final reports" (Hiebert (in progress), and Magee (in press); see also Lamberg-Karlovsky & Potts, 2001), analysis of the materials continues as opportunities allow. Metal artefacts, most especially those made of copper and its alloys, are found at this site from the late Neolithic through the Iron Age. Archaeometallurgical analysis, radiocarbon chronologies, and archaeological interpretation allow one to state when and how a type of metal or a style of object was invented, its use as a trade item, and its function and value to an ancient community. In the hopes of establishing a framework for future archaeometallurgical studies, most of the metal artefacts from Tepe Yahya, Iran, stored in the Peabody Museum of Harvard University, were analysed for elemental composition to complement stylistic and metallographic data.

Keywords: TEPE YAHYA, NATIVE COPPER, ARSENICAL COPPER, TIN BRONZE, BRASS, ICP-MS.

Introduction

A complete review of the metallurgical history of the Iranian Plateau is not only beyond the scope of this paper but made redundant in light of the excellent synthesis presented by Pigott (1999). The initial production of native copper objects begins as a localized craft in Anatolia in the late 8th millennium BCE and continues, relatively unchanged, until the Chalcolithic (5500–3200 BCE). The 5th millennium BCE on the Iranian Plateau witnesses the transition from the use of pure native copper to the smelting of copper ores chosen for their natural impurities. The most important of these impurities was undoubtedly arsenic. The Anarak-Talmessi-Meskani region in Iran contains deposits of native arsenical copper and copper-arsenides, such as domeykite and algodonite, which when melted with native copper in a crucible will dissolve "like sugar in water" to produce arsenical copper (Heskel & Lamberg-Karlovsky, 1980: 86; Pigott, 1999: 78). The melting of such ores may have led to the invention of crucible smelting as seen in the fifth millennium at Tal-i Iblis (Caldwell, 1968) and as early as the 6th millennium at Tepe Ghabristan (Majidzadeh, 1979). The precocious beginnings of smelting on the Iranian Plateau may explain the early appearance of copper-base alloys in this region relative to the rest of the Near East.

Pigott (1999: 3) outlines the four methods that may have been used to create early copper-base alloys (primarily arsenical copper) in Iran. The first method is the direct addition of different metals or metallic ores (e.g., copper-arsenides) to melted copper as mentioned above. The second method is the reduction of antimony-bearing copper-arsenates, which has been shown to produce Cu-As-Sb at low temperatures (700–900°C) (Budd *et al.*, 1992: 681). The third possibility is the reduction of roasted sulfarsenides, which produces the poisonous fume arsenious trioxide as a by-product (Lechtman & Klein, 1999: 498). Finally, arsenical

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copper could be made by smelting oxide and sulfide ores together in a process known as co-smelting. Experimentation has shown that co-smelting is a relatively easy (and non-toxic) way to create alloys with 5–10% As or Sn (Rostoker & Dvorak, 1991; Lechtman & Klein, 1999). The cementation process, whereby a primary or secondary ore containing the desired element (e.g., zinc) is roasted to oxide form and then heated with metallic copper and charcoal in a crucible, may also have been used to produce the various alloys of copper (Pollard & Heron, 1996). While it is unclear which method was first utilized on the Iranian Plateau, the amount of slag around *in situ* crucible furnaces at Tal-i Iblis and Tepe Ghabristan is suggestive of co-smelting operations and requires further investigation (Pigott, 1999: 77).

After the invention of smelting and alloying, arsenical copper remained the most common metal on the Iranian Plateau until the Iron Age (Pigott 1999: 81). Copper-tin alloys first appear in the 4th millennium BCE as evidenced by two pins and a flat axe from Necropolis A at Susa (with 0·4%. 0·8%, and 2·3% Sn respectively), a pin from Susa B (5·3% Sn), two pins from Tepe Sialk III (0·95% and 2·5% Sn respectively) and an axe from Mundigak III_6 (5% Sn) (Cleuziou & Berthoud, 1982). Although these percentages are low relative to later tin bronzes, they are significant enough to be considered intentional and not merely impurities within the copper ore. The location of the tin ore used to make these items has eluded archaeologists for decades, although most scholars agree that Afghanistan is the most probable source for early tin (Pigott, 1999). It is to be noted that there are considerable archaeological ties between the Iranian Plateau and Bronze Age sites in the Persian Gulf, but these ties do not seem to be shared in the use of tin bronzes. Thus, the extensive use of tin bronze at Tell Abraq in the United Arab Emirates, as noted in the analyses of Weeks (1999), is not complimented by the presence of much tin bronze at Tepe Yahya. This inevitably raises the debate about the supposed superiority of tin bronze over arsenical copper, with Northover (1989: 113) arguing that tin bronze can be worked harder than arsenical copper thus making it more desirable, while Lechtman (1996: 502) focuses on the ductility and working ease of copper-arsenic alloys.

The Bronze Age (3200–$\sim$1400 BCE) was a period of experimentation in metallurgical styles and compositions as busy trade routes converged on the Iranian Plateau bringing foreign influences and materials. The rise of literate and organized societies led to the production of and trade in chlorite bowls (Kohl, 1978), lapis lazuli (Herrmann, 1968), and metal ores and objects (Pigott, 1999) that were highly prized throughout the ancient Near East. Perhaps the most significant example is the tin, lapis lazuli, and gold trade from southern Bactria to Mesopotamia through sites such as Tepe Hissar and Shahr-i Sokhta (Pigott, 1999: 83). The end of the Bronze Age in the second half of the 2nd millennium was marked by the collapse of almost every major site in Iran followed within a few centuries by the introduction of iron (Dyson, 1973; Pigott, 1980).

Between 1977–1981, Dennis Heskel (1982) analysed 150 artefacts from Susa, Tepe Hissar, Shahr-i-Sokhta, and Tepe Yahya metallographically and with semi-quantitative emission spectroscopy. In this dissertation, Heskel suggests an alternative evolution of Bronze Age metallurgy, in which copper-arsenides (from the Anarak-Talmessi region) were utilized at an early stage (both smelted and possibly co-smelted) and arsenical copper remained the dominant metal type even after the introduction of tin bronze. In a resulting publication (Heskel & Lamberg-Karlovsky, 1980), the chronological sequence of metal artefact types at Tepe Yahya was explored as a model for the evolution of metallurgy on the Iranian Plateau. Unlike larger sites such as Susa, Tepe Hissar, or Shahdad, there is no evidence for smelting and little evidence of metallurgical technology at Tepe Yahya. This suggests that in the Chalcolithic, *c.* 5500–3200 BCE, when the Iranian Plateau made the shift from a localized native copper technology to more complex methods of production, metal began to arrive from metal-producing sites either as finished products or as ingots. Based on this assumption, a full elemental analysis of 105 copper-based artefacts from Tepe Yahya was undertaken in the hopes that new light might be shed on (a) the metallurgical transition from the Chalcolithic to the Bronze Age, (b) the importance of different alloys in the Bronze Age, and (c) the differences, if any, in the production of copper-base metals after the introduction of iron. As the title of this article indicates, almost all of the artefacts analysed were small functional and/or decorative items: pins, awls, needles, and jewellery. Thus, the conclusions of this study are inherently limited.

Archaeological Background

Excavations at Tepe Yahya revealed a number of distinct phases of occupation. These have been divided into periods based on architectural and ceramic stratigraphy. The first phase, known as the "Yahya" phase, is identified as Period VII (5500–4500 BCE) and incorporates a Neolithic farming village consisting of small uniform houses and characterized by a "Chaff-Tempered Coarse Ware" (Lamberg-Karlovsky & Beale, 1986). Small objects of native copper (four cylindrical pins, a sphere-headed tack, and a small corroded bead) are present in the earliest levels of Tepe Yahya. In the subsequent Period VI-VC (4500–3600 BCE) there are very few metal objects, but the first artefact with a rectangular transverse section (an awl or nail) comes from the end of the fifth millennium (Period VIA).

Period VB-VA (3600–3200 BCE) is distinguished ceramically by the presence of "Black-on-Red" ware

(found also at Tal-i Iblis to the north and Chah Husaini to the east), and by an increase in exotic goods including lapis lazuli and diatomaceous limestone from Oman (Lamberg-Karlovsky & Beale, 1986: 266). The metal collection from this period is comprised of pins (both simple and decorated), awls, and the first true needles (with eyes). The first large metal tools date from the end of this period including the spatula and "wood chisel" examined by Tylecote & McKerrell (1986). Around 3200 BCE, Tepe Yahya is abandoned without signs of violence or disruption for reasons that remain unknown.

The most striking aspect of Period IVC at Tepe Yahya (3100–2900 BCE) is the multi-roomed complex containing Proto-Elamite tablets, cylinder seals, bevelled-rim bowls, and Jemdet Nasr ceramics that dominates most of the site (Lamberg-Karlovsky & Potts, 2001). While there are a few nondescript domestic buildings on the north side of the mound, most artefacts (including most of the metal objects) are found within the Proto-Elamite complex. The metal corpus from Period IVC consists mainly of utilitarian objects plus several pieces of ore and what may be casting slag. Following this period, there is a significantly long abandonment of the site.

Period IVB (2400–2000 BCE) constitutes the period of resettlement and consists of a large complex of single-unit houses and a major chlorite bowl production workshop (*c.* 2200 BCE) (Lamberg-Karlovsky & Potts, 2001; Lamberg-Karlovsky, 1977). Although this period contains evidence for contacts with the Persian Gulf, Central Asia, and Baluchistan, the metal collection from all phases of Period IVB is unimpressive. A well-preserved shaft-hole axe was found on the floor of one room from the beginning of this period, but it remains in Iran and has not been analysed. There is some rare evidence of casting (a piece of melting overflow or "splash"), although no activity areas involved with metallurgical technology were uncovered.

The period immediately following IVB (Period IVA: 1800–1400(?) BCE) has been described as "the most successful political, social, and economic adaptation . . . at Tepe Yahya" (Lamberg-Karlovsky, 1977: 43). The majority of the ceramics of this period are unpainted and without parallel on the Iranian Plateau with the exception of the cemetery at Shahdad (Heskel & Lamberg-Karlovsky, 1980: 253). A large collection of metal objects was recovered from this period, comprised of luxury items (jewellery and pins) and functional tools (chisels, awls, knives, and drills). Most of the luxury items (including bracelets, earrings, and pendants) were recovered from an area of large, well-built houses on the south side of the mound (A- and B-contexts), while the more utilitarian items come from the north side of the mound (X-contexts). Toward the middle of the 2nd millennium BCE, Tepe Yahya (as witnessed on a number of other sites on the Iranian Plateau) is abandoned.

The Iron Age (Yahya Period III) begins *c.* 800 BCE with the establishment of a settlement probably independent of any foreign administration (Magee, in press). This period is defined by two administrative or civic buildings separated by a water channel and surrounded by a wall. At the end of the 6th century BCE, just as Cyrus and Cambyses conquered Persia and began to expand the Achaemenid Empire, the two buildings at Yahya were abandoned and two large platforms were constructed directly over them (Lamberg-Karlovsky & Magee, 1999). The Yahya platforms are similar to the mud-brick platforms from Tel-i Zohak (Magee, in press). The presence of these platforms is interpreted by Peter Magee as signifying the presence of a local rebellion against the increasing power of the Achaemenid Empire. This theory is bolstered by the fact that around the beginning of Period II at Tepe Yahya (*c.* 500 BCE), southeastern Iran was the seat of two major revolts against the Persian Empire. Such revolts are suggested in the Behistun inscription and Greek sources (Magee, in press). The collection of nonferrous metals from Periods III and II is mainly comprised of pins, awls, and needles, which show few stylistic differences from their predecessors.

Method

The entire collection (105 artefacts) of nonferrous metals in the Peabody Museum was sampled and analysed on a VG-Excell Inductively-Coupled Plasma Mass Spectrometer (ICP-MS) for 11 elements (As, Sn, Pb, Zn, Sb, Ni, Ag, Bi, Mg, Co, and V). The detection of Ca, S, and Fe was attempted, but accurate data could not be obtained due to isobaric interference with the argon carrier gas. Other elements detected by the ICP-MS include Cr, Mn, Se, Sr, Te, and Au, none of which showed great variability within the collection and thus were not included in the table. Five milligrams of corrosion-free metal was drilled or cut from each artefact and dissolved into a solution of 10 ml HNO_3 and 10 ml HCl. Each solution was then diluted by a factor of 100–200 in order to be within the detection limit of the instrument, which can detect optimally in parts per trillion. The collection was analysed multiple times and it was discovered that concentrations <100 p.p.m. varied considerably. Therefore, 100 p.p.m. was designated as an arbitrary limit for trace elemental significance in this study. Any artefacts with over 3·5% of an element besides copper were analysed on the electron microprobes (EMP's) at Harvard University and MIT, and the results of major/minor elements were used in place of the ICP-MS data (shown in **bold** in Table 1). Two artefacts were X-rayed at the Straus Center of the Fogg Art Museum by Molly McNamara to look at features under the corrosion layer, and a few objects have been metallographically analysed at the Center for Material Research in

Table 1. Chemical composition of the Tepe Yahya artifacts in chronological sequence

PM no.	Context	Type	Period	As	Sn	Pb	Zn	Sb	Ni	Ag	Bi	Mg	Co	V
359	C.71.T1.1	Hook	VII	641·767	n.s.	n.s.	126·506	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	347·71
364 ×	D.73.7.43	Tack	VII	552	n.s.	n.s.	673·2	n.s.	656·8	n.s.	n.s.	n.s.	n.s.	459·73
365	D.73.7.43	Pin	VII	257·545	n.s.	n.s.	n.s.	n.s.	423·34	n.s.	n.s.	n.s.	n.s.	460·85
396*	XC.73.3	Pin	VII	662·903	n.s.	n.s.	211·290	n.s.	404·83	202·41	n.s.	n.s.	n.s.	733·64
363 ×	D.68.4.2	Awl	VIA	14297·0	n.s.	458·217	245·940	457·02	125·14	969·90	123·96	n.s.	n.s.	454·19
357*	C.69.7.15	Pin	VC	551·683	n.s.	n.s.	n.s.	n.s.	n.s.	122·37	n.s.	n.s.	n.s.	719·12
394*	XC.71.8b.40	Pin	VB	31651·4	n.s.	n.s.	n.s.	n.s.	n.s.	669·70	n.s.	n.s.	n.s.	958·94
356*	BW/CW.71.12.1	Pin	VB-VA	36755·4	n.s.	779·324	n.s.	567·39	1551·4	5328·0	n.s.	374·42	n.s.	1032·1
358*	C.69.surf.8	Pin	VB-VA	9114·68	n.s.	947·686	n.s.	151·71	n.s.	837·02	n.s.	n.s.	n.s.	546·62
343*	B.71.18	Awl	VA	26460	n.s.	864	n.s.	18104	410·8	480	141·6	n.s.	n.s.	800·61
387	XC.70.8-1	Needle	VA	8358·87	n.s.	1614·51	n.s.	100·80	174·19	543·03	n.s.	n.s.	n.s.	660·02
390	XC.70.T1.7-1	Pin	VA	8764·94	n.s.	1032·66	655·378	133·86	478·88	1824·7	n.s.	n.s.	n.s.	510·33
404	XCE.71.T2.14.36	Pin	VA	21778·2	n.s.	3517·33	n.s.	1960·0	n.s.	424·59	n.s.	n.s.	n.s.	845·91
407	XCE.73.T1.2	Needle	VA	13401·9	n.s.	976·633	145·742	100·19	n.s.	1426·9	n.s.	n.s.	n.s.	683·12
408*	XCE.73.T2.3	Awl	VA	11048	n.s.	149·2	n.s.	636	n.s.	1220	108·4	n.s.	n.s.	691·07
362	CW.73.surf	Point	VA-IVC	16628·4	209·881	398·814	116·205	183·39	280·63	219·36	n.s.	737·17	n.s.	459·75
393·2*	XC.70.T2.2D	Pin	VA-IVC	30028·1	n.s.	1022·08	n.s.	1859·0	n.s.	235·34	n.s.	383·99	n.s.	944·31
340*	B.70.20	Pin	IVC	23700	n.s.	858·8	n.s.	474·4	4312	3768	226	n.s.	n.s.	449·48
342	B.71.11.1 surf	Awl	IVC	22402·4	n.s.	2533·60	645·070	261·56	245·87	1145·6	n.s.	n.s.	n.s.	612·12
350	B-BW.71.1.3	Hook	IVC	10277·1	333·333	4172·69	648·192	138·95	2687·9	2618·4	n.s.	n.s.	166·81	833·08
351	B-BW.71.1.3	Awl	IVC	33120·9	883·064	1731·45	648·387	136·69	2637·0	1574·1	n.s.	n.s.	126·86	859·77
384	XBE.73.T1.4.39	Pin	IVC	10812·7	239·043	607·569	n.s.	264·54	3318·7	1466·9	n.s.	n.s.	215·56	360·91
385	XBE.73.T1.surf	Stylus	IVC	2709·56	n.s.	n.s.	n.s.	n.s.	n.s.	185·65	n.s.	n.s.	n.s.	441·79
386*	XBE.73.T2.2.29-30	Pin	IVC	**48040**	n.s.	n.s.	111·776	n.s.	890·21	138·12	n.s.	n.s.	n.s.	615·12
389*	XC.70.T1.7	Axe	IVC	2545·45	n.s.	218·577	1190·11	n.s.	186·56	1039·5	n.s.	320·61	n.s.	625·07
395*	XC.71.T2.2.5	Tack	IVC	7247·48	n.s.	n.s.	n.s.	401·60	n.s.	674·84	n.s.	n.s.	n.s.	887·60
336	B.70.11	Awl	IVB2	**57996·8**	n.s.	1688·88	n.s.	101·01	353·93	833·93	n.s.	n.s.	n.s.	804·52
337*	B.70.13	Pin	IVB2	8131·73	n.s.	1101·79	n.s.	223·15	461·47	n.s.	112·57	410·17	n.s.	1087·0
338	B.70.15	Pin	IVB2	1836·72	n.s.	222·754	n.s.	2930·9	n.s.	7069·8	117·76	n.s.	n.s.	698·89
339·1	B.70.15.3	Pin	IVB2	37552	n.s.	n.s.	n.s.	n.s.	2172	n.s.	n.s.	n.s.	n.s.	720·44
339·2	B.70.15.3	Pin	IVB2	32549·4	n.s.	n.s.	n.s.	n.s.	2013·7	n.s.	n.s.	—	—	—
341	B.70.T3.1	Awl	IVB2	9189·51	117·338	n.s.	140·725	109·27	1685·0	2905·6	n.s.	n.s.	109·87	657·28
344	B.71.4.2	Pin	IVB2	**16100**	**4000**	**2900**	**0**	**600**	**1400**	**800**	—	—	**100**	—
345	B.71.4.3	Pin	IVB2	26455·6	n.s.	1572·58	552·419	n.s.	n.s.	2447·5	n.s.	n.s.	n.s.	816·12
346	B.71.7	Flat wire	IVB2	8108·43	4389·55	387·951	n.s.	n.s.	6919·6	n.s.	n.s.	n.s.	1867·9	841·44
348·1	B-BW.70.T4.8	Pin	IVB2	754·929	n.s.	158·148	n.s.	n.s.	n.s.	785·91	n.s.	385·58	n.s.	875·70
348·2	B-BW.70.T4.8	Pin	IVB2	32784·7	n.s.	2248·69	617·706	4225·3	304·62	4217·3	n.s.	n.s.	n.s.	879·84
349	B-BW.70.T4.9	Needle	IVB2	7628·74	n.s.	341·716	629·141	n.s.	1536·9	3712·5	n.s.	n.s.	n.s.	796·92
353*	BW.69.T5.5	Needle	IVB2	29364	n.s.	211·2	540	244·4	828	306·4	n.s.	n.s.	n.s.	895·62
355	BW.69.T6.2	Pin	IVB2	417·670	n.s.	n.s.	n.s.	n.s.	n.s.	1621·6	n.s.	n.s.	n.s.	590·44
376	BW.69.T6.1	Pin	IVB2	29338·6	n.s.	141·883	n.s.	n.s.	870·94	431·66	n.s.	n.s.	n.s.	774·98
393·1*	BW.69.T5.9	Awl	IVB2	26689·3	n.s.	333·867	n.s.	155·51	785·17	1764·3	n.s.	n.s.	n.s.	791·89
414·1*	BW.69.T5.5	Ring	IVB2	7280	3788	312·8	n.s.	n.s.	6828	161·2	n.s.	203·48	542·57	216·13
323 ×	ANE4.70.T5.1	Splash?	IVB	23840·9	n.s.	6787·27	616·302	n.s.	1510·9	469·18	193·63	616·22	n.s.	872·27
318	AN2.73.14	Awl	IVB1	15543·4	n.s.	n.s.	255·757	218·98	151·51	129·29	n.s.	n.s.	n.s.	690·00
334	B.70.1.4	Pin	IVB1	30265·3	n.s.	796·039	n.s.	118·81	1348·9	n.s.	n.s.	n.s.	n.s.	796·43
335*	B.70.1.4	Fragment	IVB1	6562·24	n.s.	n.s.	783·132	n.s.	4847·3	n.s.	n.s.	n.s.	356·54	873·96
347*	B.73.2.6	Spatula	IVB1	31092	n.s.	632·4	773·2	n.s.	1437·2	115·2	n.s.	n.s.	n.s.	583·93
388	XC.70.T1.6	Pin	IVB1	22113·1	n.s.	193·131	n.s.	445·65	115·95	1846·7	n.s.	377·86	n.s.	744·44
392*[1]	XC.70.T2.2B	Bead	IVB1	21074·1	n.s.	6505·01	339·879	112·62	773·94	271·34	n.s.	n.s.	n.s.	1030·7
316A	AN1-ANW1.71.1	Pin-shaft	IVA	10519·1	**29,000**	195·573	n.s.	200	742·05	n.s.	n.s.	n.s.	n.s.	998·80
316B	AN1-ANW1.71.1	Pin-wrap	IVA	6052·10	29891·7	296·993	n.s.	270·94	290·18	513·02	105·41	1343·0	n.s.	1351·4

316C ×	AN1-ANW1.71.1	Pin?	IVA	10583·3	**66420**	**106090**	n.s.	1306·3	857·14	1539·6	1283·3	n.s.	121·89	839·98
317 ×	AN1-ANW1.71.2	Bangle	IVA	—	**~25%**	**~75%**	—	—	—	—	—	—	—	—
324	ANW1.70.1	Ribbon	IVA	1346·39	2595·21	3336·65	**170100**	148·20	2094·0	185·25	n.s.	n.s.	175·52	794·39
325	ANW1.70.5	Spatula	IVA	3255·53	n.s.	n.s.	655·935	n.s.	847·88	706·63	n.s.	n.s.	n.s.	436·01
326 ×	ANW1.71.3.4	Bracelet	IVA	4524·85	n.s.	8560·63	**193600**	1042·9	243·33	518·09	n.s.	n.s.	n.s.	551·55
327 ×	AW.70.8>9	Fragment	IVA	498·023	7794·46	18181·8	**169000**	1077·4	420·15	297·62	n.s.	n.s.	n.s.	660·63
332·1	B.68.T1.10	Awl	IVA	39377·7	5187·87	199·595	n.s.	n.s.	1626·6	250·50	n.s.	n.s.	n.s.	824·26
333*	B.69.2.1	Pin	IVA	4708	12480	n.s.	n.s.	n.s.	463·6	332·8	n.s.	1063·3	n.s.	1120·5
367	XB.70.T2.3	Awl	IVA	16339·2	n.s.	313·981	n.s.	112·14	559·77	1001·0	n.s.	n.s.	n.s.	783·46
368	XB.70.T2.5A	Pin	IVA	34300·9	n.s.	n.s.	n.s.	n.s.	205·94	n.s.	n.s.	n.s.	n.s.	246·97
371	XB.71.T1.7	Pin	IVA	20293·0	n.s.	1609·50	602·772	325·14	1135·8	556·43	n.s.	n.s.	n.s.	610·97
373	XB.71.T2.6b	Pin	IVA	9428·57	**83320**	12390·3	n.s.	435·41	2351·3	1280·8	n.s.	412·21	n.s.	260·06
374	XB.73.2	Pin	IVA	144·466	n.s.	n.s.	740·442	n.s.	1476·8	523·13	n.s.	n.s.	n.s.	376·96
375	XBE.71.T1.5	Pin	IVA	36780·8	358·565	122·709	n.s.	n.s.	n.s.	617·52	n.s.	n.s.	n.s.	557·17
377	XBE.71.T2.3.1	Pin	IVA	36594·3	n.s.	n.s.	n.s.	n.s.	n.s.	447·38	n.s.	n.s.	n.s.	868·33
378	XBE.71.T2.3b.2	Bangle	IVA	5431·45	**76200**	1029·43	n.s.	256·45	3153·6	242·74	420·16	n.s.	185·57	1064·6
379	XBE.71.T2.5	Bracelet	IVA	39100·8	n.s.	n.s.	n.s.	n.s.	5717·7	n.s.	n.s.	n.s.	1136·5	760·05
382*	XBE.73.T1.1	Pin	IVA	3785·94	**86640**	1299·59	n.s.	126·90	3404·4	126·50	n.s.	n.s.	159·61	970·97
383	XBE.73.T1.1	Awl	IVA	5183·83	1446·46	668·686	345·858	127·27	4101·0	n.s.	n.s.	n.s.	666·35	571·72
391	XC.70.T1.8B-1	Pin	IVA	**68140**	n.s.	584	n.s.	n.s.	3920·4	230·8	n.s.	n.s.	n.s.	815·62
397	XCE.71.T1.12b	Engraver	IVA	20172·6	n.s.	1774·69	n.s.	n.s.	375·90	205·22	n.s.	n.s.	n.s.	796·46
398	XCE.71.T1.13c	Drill	IVA	18547·2	n.s.	809·657	n.s.	1042·2	n.s.	2596·3	n.s.	n.s.	n.s.	766·86
399	XCE.71.T1.2.1	Pin	IVA	17028·2	n.s.	7068·54	n.s.	806·45	n.s.	1669·3	166·12	n.s.	n.s.	700·36
400	XCE.71.T1.surf	Awl	IVA	25215·1	n.s.	1245·81	107·569	n.s.	1194·0	102·78	n.s.	n.s.	n.s.	837·21
401	XCE.71.T1.surf	Awl	IVA	33188	n.s.	369·6	n.s.	926·4	202·8	767·2	120·8	n.s.	n.s.	846·89
402	XCE.71.T1.surf	Nail/awl	IVA	22656·0	n.s.	2217·49	1988·07	292·64	349·90	506·56	n.s.	n.s.	n.s.	1009·7
403	XCE.71.T1.surf	Needle	IVA	36732·6	n.s.	n.s.	n.s.	n.s.	n.s.	209·10	n.s.	390·66	n.s.	1205·4
405	XCE.71.T2.2	Awl	IVA	28629·0	n.s.	n.s.	n.s.	6052·4	693·54	172·17	n.s.	381·00	n.s.	1224·8
406	XCE.71.T2.8	Stylus	IVA	14238·0	1205·15	2614·28	n.s.	494·84	2976·5	1152·3	n.s.	n.s.	139·40	815·51
413*	XBE.73.T1.2	Flat frag	IVA	20885·3	n.s.	945·059	182·213	621·34	129·64	260·47	346·64	n.s.	n.s.	850·83
414·2*	XBE.73.T2.2	Ring	IVA	15462·1	n.s.	3294·82	n.s.	n.s.	1136·6	200·79	n.s.	201·05	n.s.	198·93
415*	XBE.73.T1.10	Flat frag	IVA	18953·7	n.s.	n.s.	n.s.	2595·5	5501·0	1939·6	n.s.	199·22	n.s.	217·57
321	AN4.71	Pin	III	11177·4	n.s.	690·322	177·822	n.s.	4133·0	n.s.	n.s.	n.s.	242·15	688·66
322	AN4.71.2	Awl	III	5115·07	6813·49	307·936	n.s.	n.s.	667·06	225·39	n.s.	n.s.	n.s.	746·24
352*	BW.69.T5.4	Splash	III	13061·3	7544·55	2067·32	163·168	155·64	3931·8	305·34	n.s.	243·11	207·25	919·87
302 ×	A.71 dump	Wand	II	1046·61	11749·0	4717·13	2523·10	204·78	1595·2	**~60%**	203·18	n.s.	n.s.	1361·8
303	A.71.1.2	Spatula	II	23422·3	n.s.	2646·21	n.s.	1023·5	182·07	443·82	121·11	n.s.	n.s.	800·47
304	A.71.2	Pin	II	**51368·2**	n.s.	187·070	674·747	n.s.	1571·7	1319·1	n.s.	n.s.	n.s.	1034·1
305	A.71.24	Fragment	II	14842·7	n.s.	n.s.	645·161	n.s.	1447·5	158·87	n.s.	n.s.	n.s.	787·93
308	A3E.70.T3.70	Needle	II	18400	n.s.	317·979	647·676	n.s.	2300·2	503·03	n.s.	370·61	n.s.	1110·6
310	A-AN.71.14	Awl	II	12321·2	n.s.	4915·66	n.s.	764·25	339·75	n.s.	1082·3	n.s.	n.s.	965·85
311	A-AN1.71.14.2	Pin	II	24440·8	n.s.	6044·08	n.s.	4260·5	446·49	1326·6	n.s.	n.s.	n.s.	908·68
312	A-AN1.71.10 surf	Fragment	II	3375	n.s.	n.s.	n.s.	n.s.	196·03	621·03	n.s.	n.s.	n.s.	791·91
313	A-AN1.71.3	Hook	II	32865·0	n.s.	n.s.	n.s.	n.s.	1158·7	n.s.	n.s.	n.s.	120·59	827·11
328	AW.70.T1.3	Pin head	II	17142·8	**41000**	2039·43	n.s.	204·42	1573·4	514·68	n.s.	n.s.	n.s.	834·04
329	AW.71.9	Pin	II	18666·6	**98800**	21232·9	175·903	540·96	672·28	1096·3	574·69	n.s.	n.s.	931·22
330	AW2.73.26.1	Hook	II	12475·0	n.s.	146·906	254·291	909·38	266·26	110·97	n.s.	n.s.	100·66	711·10
331	AW-ANW1.71.1	Pin	II	**67140·2**	n.s.	n.s.	n.s.	n.s.	154·45	1029·7	n.s.	n.s.	n.s.	857·19
300	A.68.1	Tack	S	38446·6	430·985	542·454	623·742	150·50	929·57	577·06	195·97	n.s.	n.s.	1034·6
301	A.68.1.3	Awl	S	1140	2188	4544	564	338	456	560	n.s.	n.s.	n.s.	806·03
409	XD.70.surf	Hook	S	38500	n.s.	1421·37	125·403	n.s.	194·75	168·54	n.s.	n.s.	n.s.	846·32
410	XD.70.surf II	Pin	S	387·649	n.s.	n.s.	665·338	n.s.	363·34	113·14	n.s.	n.s.	n.s.	825·82
411	A.71.10	Spatula	S	13620·2	253·333	110·707	n.s.	306·26	101·41	162·42	n.s.	n.s.	n.s.	928·01

All values in parts per million (10,000 p.p.m. = 1% by weight) unless otherwise labeled.

[1]The sample for this analysis was heavily corroded, thus Heskel's (1982) spectrographic detection of 3·4% As and 2·4% Pb is a better estimate.

*Metallographic analysis in Heskel 1982; × metallographic analysis by Thornton; **B**, electron microprobe result; —, not tested; S, surface collection; n.s., "not significant" = <100 p.p.m.

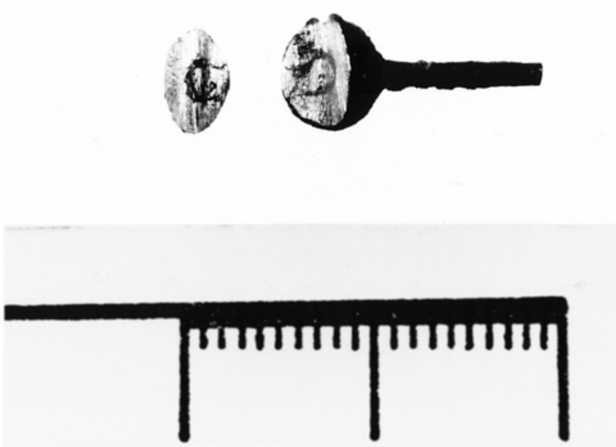

Figure 1. The Period VII tack (PM No. 364) after sampling, showing how one piece of metal was wrapped around a central shaft.

Figure 2. Photomicrograph of the deformed grains and twinning indicative of native copper, which was hammered and annealed around the central shaft to create the spheroid head of the Period VII tack (mag: 31, 8 s of $K_2Cr_2O_7$).

Archaeology and Ethnology (CMRAE) at MIT (marked with an "*" or "×" in Table 1) with the help of Heather Lechtman and Thomas Tartaron.

Results

The metal artefacts that are present as early as 5500 BCE at Tepe Yahya (Period VII) are nearly free of impurities and show little stylistic variation. The exceptional piece from this early group is the 1·8 cm-long tack with spheroid head (PM No. 364) from *c.* 5000 BCE. Metallographic analysis shows how this artefact was made by hammering and annealing a small rod of native copper around the upper end of a delicate shaft of native copper 1 mm in diameter. A nail/awl (PM No. 363) from the VIA period (*c.* 4200 BCE) is the first item with a significant impurity (1·43% As) and the first item with a rectangular cross-section. Metallographic analysis of this object shows corroded and deformed dendrites, not the large and small grains with long, thin twinning indicative of untreated native copper. The fact that this object is contemporary with the early crucible smelting at Tal-i Iblis (~150 km to the north) should not be ignored. A pin (PM No. 357) from a later stratigraphic layer (Period VC) than the cast nail/awl is made of native copper that is chemically and microstructurally identical to the native copper artefacts from Period VII (Heskel, 1982: 76) (Figures 1 and 2).

The production of native copper artefacts at Yahya is eventually replaced in Period VB (3600–3400 BCE) by a more complex metallurgical technology. This change was first observed by Heskel's metallographic analysis of a knob-headed pin (PM No. 394) that had been cast, worked, and annealed (Heskel & Lamberg-Karlovsky, 1986: 212). Our ICP-MS analysis also demonstrates a change in the metallurgy from periods VC to VB as chemically-pure copper disappears and arsenical copper "trinkets" (i.e., pins, needles, awls, etc.) of 1–4% As predominate. It is notable that the earliest copper ores and a single ingot found at Yahya all date from this period. This transition is marked by the appearance of significant trace elements in most objects, most notably an awl (PM No. 343) with 1·8% Sb, which may indicate low-temperature reduction of copper arsenates (Pigott, 1999: 88).

The metal artefacts from the Proto-Elamite compound of Period IVC (3100–2900 BCE) vary little from those of the earlier Period VB-VA. Copper-arsenic alloys (with 1–5% As) remain constant, although the types of objects become more varied as evidenced by a U-shaped pot handle (?), a stylus, and a flat axe that was analysed by Heskel (1982: 76). This axe (PM No. 389) showed no evidence of edge hardening and was made from almost pure copper (2545 p.p.m. As is the greatest impurity), which is an extremely soft material when not work-hardened. The chemical composition combined with the lack of edge-hardening suggests that this was a decorative rather than a functional axe.

Despite a break in habitation after Period IVC, the metal objects of Period IVB are stylistically and chemically similar to their predecessors. Arsenical copper (with 1–6% As) predominates, although "pure" copper artefacts are still present (e.g., PM No. 355) and may reflect a continued use of local native copper resources. The presence of ~0·4% Sn in three objects is surprising given the complete lack of tin in preceding periods, and may indicate the use of tin-rich copper ores, the recycling of low-tin bronzes, or the addition of cassiterite as a flux (cf. Rapp, 1988; Muhly, 1999).

The most interesting item in the collection from Period IVB is the incised and perforated X-shape bead (PM No. 392) analysed by Heskel (1982: 86–87). His spectroscopic analysis detected significantly more arsenic and lead (3·4% As and 2·4% Pb) in this artefact than our ICP-MS analysis, a discrepancy that was

probably caused by the highly-corroded state of the ICP-MS sample. Thornton (2001, unpub.) has shown that Heskel's detection of arsenic in all artefacts was consistently too low by 1–2% due to the inherent limitations of this method at the time (see also Heskel, 1982: 284–285), which may suggest that the real arsenic content of this bead is closer to 5%.

The terminal Bronze Age at Tepe Yahya, Period IVA, contained the largest number of metal artefacts and the greatest stylistic and chemical variation. The functional objects from the north side of the mound (X-contexts) were mainly tools and the standard "trinkets" made of arsenical copper (1–7% As). Berthoud's (1979) analysis of a single piece of slag from this area revealed high levels of arsenic (6% As), which suggests that copper-arsenic alloys were being cast at Yahya to some degree in this period. Notable exceptions in this area of the site include a pin (PM No. 373) with 8·33% Sn, a bangle (PM No. 378) with 7·62% Sn, and the pin with incised head (PM No. 382) with 8·66% Sn that was analysed metallographically by Heskel (1982: 96–97). Electron microprobe analysis (EMPA) revealed significant S and/or Fe impurities (0·1–0·7%) in the tin bronzes of this period, concentrations not found in any other artefacts analysed by this method.

The metal objects from the south side of the mound (A- and B-contexts) have little precedence at Tepe Yahya or, for that matter, on the entire Iranian Plateau. Artefact No. 316 is comprised of two parts: a twisted pin (PM No. 316A) of 2·9% Sn with the grooves wrapped in a similar low-Sn bronze. The wrap material (No. 316B) was extremely corroded and may in fact have had more than 3% Sn. Artefact 316C (originally thought to be part of 316A and B) is a fragment of a pin or a bracelet containing 10·61% Pb and 6·64% Sn. Metallographic analysis as polished shows extremely large lead inclusions, and EMPA found numerous high-Sn eutectic microconstituents. What makes this artefact more intriguing is that it was found next to a bangle (PM No. 317) containing roughly 75% Pb and 25% Sn (as estimated from EMPA rastering). Whether artefact No. 316C was made at Yahya using "proto-pewter" such as artefact No. 317 is unclear, but their discovery together seems more than coincidental.

Artefact No. 325—one of four miniature "spatulas"—is unique in this area of the mound because it is almost pure copper. It may be indicative of the type of copper used as a base metal to produce these other heavily-alloyed artefacts (none of which contains significant trace elements as impurities). Artefacts No. 332·1 and 333 are interesting because they are the only standard "trinkets" found on the south side of the site in Period IVA, yet they are distinguished from their northern counterparts by the presence of significant tin (0·5% and 1·25% Sn respectively). These two items are contextually related to the tin bronze dagger (8·9% Sn) analysed by Tylecote & McKerrell (1986) and the BMAC-style compartmented seal that remains in Teheran (see Hiebert & Lamberg-Karlovsky 1992: 6).

Figure 3. The brass bracelet of Period IVA (PM No. 326) after sampling. X-ray analysis showed a strain fracture throughout the object and no features under the corrosion.

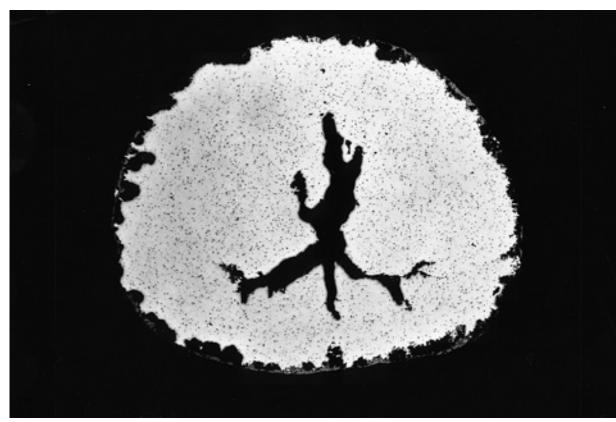

Figure 4. Photomicrograph of the as-polished transverse section of the brass bracelet from Period IVA with the central strain fracture. Note the oblong shape of the section relative to the brass fragment (see Figure 6) (mag: 21).

The most unanticipated artefacts from this area of the site are the three brass jewellery fragments (PM No. 324, 326, and 327) dated to the 15th century BCE. Their bright golden colour and the presence of 17–20% Zn (as determined by EMPA) are enough to dispel any suggestion of accidental production. Metallographic analysis of one bracelet fragment (PM No. 326: 19·4% Zn, 0·9% Pb) revealed a large strain fracture that runs through the centre of the entire object (as shown by X-ray analysis) (Figures 3 and 4). Etching a mounted transverse section with $K_2Cr_2O_7$ revealed equiaxed grains that are small on the edge of the sample and larger towards the middle fissure. The other fragment, artefact No. 327 (16·9% Zn, 1·8% Pb), is more circular in cross-section than artefact No. 326, and etching with $K_2Cr_2O_7$ showed uniform small grains from edge-to-middle and a V-shaped fissure on one edge (Figures 5 and 6). The analysis suggests that this artefact was originally a rod of rectangular cross-section that was

Figure 5. The brass fragment from Period IVA (PM No. 327) with the right-hand end worked flat like a nail.

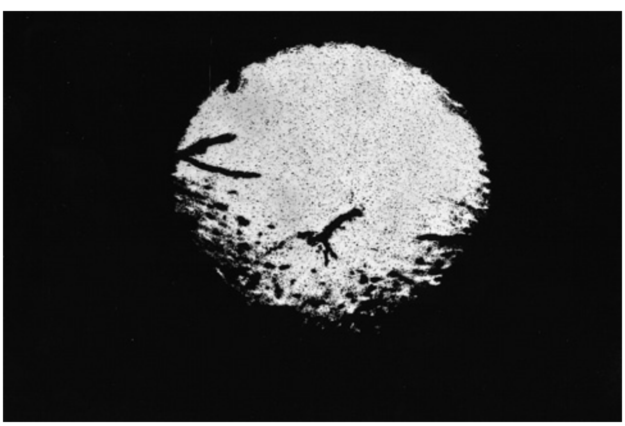

Figure 6. Photomicrograph of the as-polished transverse section of the Period IVA brass fragment. Note the V-shaped fissure on the left side where the metal was worked into a circular shape (mag: 17).

plastically deformed into its circular shape (as suggested by the V-shaped fissure), unlike artefact No. 326 that retained much of its original rectangularity. There is some question as to whether artefact No. 327 is a fragment of a bracelet or not, since the only true end of the artefact was worked flat like a nail, but too much of the artefact is missing to make a judgment.

The Iron Age copper-base metal artefacts from Tepe Yahya show little difference in style or composition from earlier periods. Copper-arsenic alloys are the most common (from 1–7% As), with two tin bronze objects (both decorative pins) and one awl of low tin (0·7%) being the exceptions. One item of interest is a silver-copper-tin "wand" (roughly 60% Ag, 39% Cu, and 1% Sn as determined by point-count analysis and EMPA) found in a deposit of fill from Period II. Metallographic analysis indicates that this object was cast and then heavily worked on both ends, one of which has two rivet holes (as shown by X-ray analysis). The purpose of this object is unknown, but it may be significant that silver-copper alloys are usually associated with elite Persian decorative artefacts.

Conclusions

The first conclusion to be drawn from our ICP-MS analysis of Tepe Yahya pins, awls, needles, and other ubiquitous items is a confirmation of Heskel and Lamberg-Karlovsky's theory that the rise of Bronze Age metallurgy on the Iranian Plateau does not conform to the early models of metallurgical evolution proposed by Childe, Wertime, and Renfrew (see Heskel & Lamberg-Karlovsky, 1980: 229). That is, the evolution of metal technology as seen at Tepe Yahya is not a linear transgression in which each new metal type replaces its predecessor because of innate superiority. The earliest metal items at this site (Period VII, *c.* 5500 BCE) are made from native copper that are in at least one case worked and annealed with great skill. At the end of the 5th millennium (Period VIA), the first cast object made of arsenical copper appears, coincident with the metallurgical installations of Tal-i Iblis and Tepe Ghabristan. The importation of cast Cu-As items to Tepe Yahya does not instantly replace the established native copper industry, as is evidenced by the native copper pin (PM No. 357) from Period VC. In fact, it is not until 300 years after the arrival of the first Cu-As object that native copper all but disappears from the archaeological record to be superseded by arsenical copper, which remains the dominant copper-base alloy for the next 3000 years.

This diffusion of metallurgical technology in the second half of the 4th millennium undoubtedly encouraged the spread of the Proto-Elamites, or at least their influence, from the resource-poor Fars region to all areas of the Iranian Plateau. The Proto-Elamite expansion in Period IVC brought to Yahya new types of metal objects, yet little variation in the composition of artefacts. Similarly, the metal artefacts from Period IVB show little change from those of Period IVC despite a 500-year gap in settlement. This may suggest that the arsenical copper "trinkets" were part of a localized "cottage" industry at Yahya that was unaffected by shifts in the sociopolitical structure of the site (cf. Heskel, 1982).

The metals from Period IVA indicate a level of visual display and success previously unmatched at this site. The objects from the northern area of the site are mainly tools (e.g., awls, an engraver, a drill, and flat razors) all made from low-As copper (1–2% As) and a number of decorative items (e.g., a bracelet, bangles, and pins) made of high-As copper (3–7% As) and tin bronze. Why the tools would contain less arsenic than decorative items is unclear, although one possibility is that the value of an object was determined by its colour, and thus the more arsenic-rich material with its silvery sheen would be desirable for decorative items. Further evidence for the importance of colour can be

Figure 7. The Ag-Cu-Sn "wand" from Period II (PM No. 302). X-ray analysis revealed two holes on the right-hand end, which was not the original end of the object.

found in the artefacts from the southern side of the mound, which range from reddish copper to golden brass to silvery proto-pewter.

The most obvious conclusion to be drawn from the analysis of copper-base metal artefacts from the Iron Age is that the introduction of iron has little effect on the production or importation of arsenical copper "trinkets". Compared to the elaborate items from Period IVA, the artefacts from Periods III and II are fairly mundane. This may add support to Magee's theory that Tepe Yahya was not an active part of the Archaemenid Empire, based on the lack of the elaborate metal objects that are indicative of Persian metallurgy. An exception is the Ag-Cu-Sn 'wand' from Period II, which is beautifully crafted and of a brilliant silver colour (Figure 7).

The three Cu-Zn objects of Period IVA are some of the oldest substantiated brasses ever found and could alter our perception of technological diffusion in the Bronze Age. Another early reported brass, a pin with supposedly 23·4% Zn, comes from the Early Bronze Age site of Gezer in Palestine *c.* 1400–1200 BCE (Craddock, 1978). Paul Craddock, perhaps the world's foremost authority on early brass, refuted the analysis of this item (done in 1912) based partly on the poor quality of the report by J. E. Purvis and partly on the analysis of a dagger from Gezer (*c.* 1500 BCE) with only 4% As, "suggesting that far from being innovators the smiths of Gezer were rather conservative" (Craddock, 1978: 3). He suggested at the time that the earliest "deliberate" brasses came from Anatolia (*c.* 1st millennium BCE) with zinc concentrations not exceeding 10%.

In the past few years, two separate projects at the CMRAE metallography labs at MIT (including the analysis of the two fragments in this study) have shed new light on the invention of brass. The first project involved nine objects (three rings, three pins, and three projectile points) from the Nuzi Collection in the Harvard Semitic Museum that were analysed metallographically by Christine Bedore Ehlers and Chris Dixon from 1996–1998 (Bedore Ehlers & Dixon, 1998). Electron microprobe analysis showed two of the rings to be Cu-Zn-Sn-Pb mixtures with zinc levels of 12·2% and 14·4%, tin levels of 6·3% and 0·4%, and lead levels of 4·73% and 3·35% respectively. Both rings could be securely assigned to an archaeological context lying underneath (earlier than) a destruction layer from 1350 BCE (Ehlers, pers. comm.).

While we must take the Gezer pin analysis with a critical eye, it would coincide nicely with the appearance of Cu-Zn alloys with high levels of zinc both in the Hurrian kingdom of Mitanni (Nuzi) and in southeastern Iran before the 14th century BCE. The fact that the Gezer blade contained 4% arsenic should not be indicative of a "conservative" metallurgy that could not include brass, since at Yahya we have brasses and other advanced copper-base alloys contemporary to mundane Cu-As tools of 1–5% As. Furthermore, neither Nuzi nor Tepe Yahya were sites of great metallurgical innovation, yet both managed to acquire one of the Bronze Age's rarest metals.

The major questions that remain are how was the earliest brass made and where did it come from? Of the two accepted methods of early brass production (smithsonite ($ZnCO_3$) and sphalerite (ZnS) cementation), the use of smithsonite is known to contribute significant amounts of iron and manganese to the resulting metal (Ponting & Segal, 1998: 117). Manganese was not a significant trace element in any of the artefacts from this collection including the three brasses (range: 0–28·5 p.p.m. Mn), and iron was not detected in the EMPA of artefact No. 324 (see Thornton, 2001). Therefore, we must conclude that the earliest brass production utilized sphalerite cementation, a method that Ponting & Segal (1998: 117) assign to northern Anatolia (*c.* 1st millennium BCE). The fact that brass first appears in Iran a few centuries before iron arrives from Anatolia may support this theory (Pigott, 1980). Alternatively, the sudden occurrences of tin, lead, and zinc alloyed with copper in an area of Tepe Yahya that shows material links with the BMAC may suggest a Central Asian origin for brass. The prologue to the history of brass is only now being written, and future analyses will hopefully shed more light on this elusive subject.

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